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U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

**TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371**

1328/3

U.S. APPLICATION NO. (REQUIRED FOR PCT/PTO)

09/7485277

INTERNATIONAL APPLICATION NO.

PCT/EP98/05030

INTERNATIONAL FILING DATE

07 August 1998 (7.08.98)

PRIORITY DATE CLAIMED

07 August 1997 (7.08.97)

-TITLE OF INVENTION

PROCESS TO MANUFACTURE A CELLULOSE FIBRE FROM HYDRATE CELLULOSE

APPLICANT(S) FOR DO/EO/US

NEUMAYR, Achim, et al.

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in **English** with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.
☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information:

15 Sheets of Informal Drawings; Copy of Form PCT/RO/101 as filed and as received from the EPO; Copy of WO 99/07926 (w/ English Abstract); Copy of Form PCT/ISA/210; Copy of Form PCT/IPEA/401; Copy of Form PCT/IB/308 (English Translation); Copy of Form PCT/IPEA/409;

Explanatory with 37 CFR 3.28 and 3.31 is included.
 Number 53059467605
 Date of Deposit 02-03-00
 I hereby certify that this paper or fee is being deposited with the United States Postal Service "express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to the Commissioner of Patents and Trademarks, Washington, D.C. 20231. Katrina A. Holland, Lilian S. Glenn, Lynette M. Bailey, Amy J. Martin, N. Ruth Reid

Lillian S. Glenn

17. ☒ The following fees are submitted:**BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5))**

Neither international preliminary examination fee (37 CFR 1.482)
nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO
and International Search Report not prepared by the EPO or JPO \$970.00

International preliminary examination fee (37 CFR 1.482) not paid to
USPTO but International Search Report prepared by the EPO or JPO \$840.00

International preliminary examination fee (37 CFR 1.482) not paid to USPTO but
international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$760.00

International preliminary examination fee paid to USPTO (37 CFR 1.482)
but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$670.00

International preliminary examination fee paid to USPTO (37 CFR 1.482)
and all claims satisfied provisions of PCT Article 33(1)-(4) \$96.00

ENTER APPROPRIATE BASIC FEE AMOUNT =**CALCULATIONS**

PTO USE ONLY

Surcharge of \$130.00 for furnishing the oath or declaration later than ☐ 20 ☐ 30
months from the earliest claimed priority date (37 CFR 1.492(c)).

\$ 840.00

\$ 0.00

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE
Total claims	53	-20 =	33
Independent claims	1	-3 =	0

\$ 594.00

\$ 0.00

\$ 0.00

MULTIPLE DEPENDENT CLAIM(S) (if applicable)

+ \$260.00

TOTAL OF ABOVE CALCULATIONS =

\$ 1,434.00

Reduction of 1/2 for filing by small entity, if applicable. A Small Entity Statement
must also be filed (Note 37 CFR 1.9, 1.27, 1.28).

\$ 717.00

SUBTOTAL =

\$ 717.00

Processing fee of \$130.00 for furnishing the English translation later than ☐ 20 ☐ 30
months from the earliest claimed priority date (37 CFR 1.492(f)).

\$ 0.00

TOTAL NATIONAL FEE =

\$ 717.00

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be
accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property

\$ 0.00

TOTAL FEES ENCLOSED =

\$ 717.00

Amount to be:
refunded \$
charged \$

- a. ☒ A check in the amount of \$ 717.00 to cover the above fees is enclosed.
- b. ☐ Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees.
A duplicate copy of this sheet is enclosed.
- c. ☐ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any
overpayment to Deposit Account No. _____. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO

Richard E. Jenkins
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3100 Tower Boulevard
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USA

SIGNATURE

Richard E. Jenkins

NAME

28,428

REGISTRATION NUMBER

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: Neumayr, Achim; Hasl, Herbert

Application No.: 09/485,277

Filed on: February 3, 2000

Title: PROCESS TO MANUFACTURE A CELLULOSE FIBRE FROM HYDRATE CELLULOSE

**STATEMENT CLAIMING SMALL ENTITY STATUS
(37 CFR 1.9(f) and 1.27(b))--INDEPENDENT INVENTOR**

As a below named inventor, I hereby state that I qualify as an independent inventor, as defined in 37 CFR 1.9(c), for purposes of paying reduced fees to the United States Patent and Trademark Office under Sections 41(a) and (b) of Title 35, United States Code, to the Patent and Trademark Office, with regard to the invention described in the application identified above.

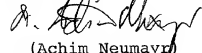
I have not assigned, granted, conveyed or licensed, and am under no obligation under contract or law to assign, grant, convey or license, any rights in the invention to any person who would not qualify as an independent inventor under 37 CFR 1.9(c), if that person had made the invention, or to any concern that would not qualify as a small business concern under 37 CFR 1.9(d), or a nonprofit organization under 37 CFR 1.9(e).

No person, concern or organization exists to which I have assigned, granted, conveyed, or licensed or am under an obligation under contract or law to assign, grant, convey, or license any rights in the invention.

I acknowledge the duty to file, in this application or patent, notification of any change in status resulting in loss of entitlement to small entity status prior to paying, or at the time of paying, the earliest of the issue fee or any maintenance fee due after the date on which status as a small entity is no longer appropriate. (37 CFR 1.28(b))

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which this verified statement is directed.

Achim Neumayr

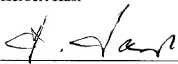


(Achim Neumayr)

Signature of Inventor

Date 25.04.2000

Herbert Hasl


Signature of Inventor

Date 25.4.2020

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Lillian S. Glenn

09/485277

420 Rec'd PCT/PTO 03 FEB 2000

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: Neumayr et al.

Examiner:

PCT Application No.: PCT/EP98/05030

Group Art Unit:

Filed: 07 August 1998 (Priority Date - 07 August 1997)

Docket No.: 1328/3

For: PROCESS TO MANUFACTURE A CELLULOSE FIBRE FROM HYDRATE CELLULOSE

PRELIMINARY AMENDMENT

Honorable Commissioner of
Patents and Trademarks
BOX PCT
Washington, D.C. 20231

Sir:

AMENDMENTS

Kindly amend the subject application as follows:

IN THE SPECIFICATION:

Page 1, before written line 1, please insert the heading --Description--.

Page 1, before written line 2, please insert the heading --Technical Field--.

Page 1, after written line 4, please insert the heading --Background Art--.

Page 1, after written line 15, please insert the heading --Disclosure of the
Invention--.

Page 3, after written line 5, please insert the heading --Brief Description of the
Drawings--.

Page 4, after written line 4, please insert the heading --Detailed Description of the
Invention--.

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IN THE CLAIMS:

Page 33, delete "Patent Claims" and please insert --That which is claimed is:--.

Please amend the claims as follows:

33. (Once Amended) Cellulose fibre, obtainable by a process in accordance with [one of Claims 1 to 32] Claim 1.

37. (Once Amended) Fabric comprising a backing fabric and a pile woven into the backing fabric which contains the fibres in accordance with [one of the Claims 33 to 36] Claim 33.

45. (Once Amended) Use of the fabric in accordance with [one of the Claims 37 to 44] Claim 37.

46. (Once Amended) Use of the fabric in accordance with [one of the Claims 37 to 44] Claim 37 to reduce the surface tension of water.

47. (Once Amended) Use of the fabric in accordance with [one of the Claims 37 to 44] Claim 37 to make textiles.

48. (Once Amended) Use of the fabric in accordance with [one of the Claims 37 to 44] Claim 37 to make clothing textiles.

49. (Once Amended) Use of the fabric in accordance with [one of the Claims 37 to 44] Claim 37 to make personal hygiene articles.

50. (Once Amended) Use of the fabric in accordance with [one of the Claims 37 to 44] Claim 37 as a particle filter.

51. (Once Amended) Use of the fabric in accordance with [one of the Claims 37 to 44] Claim 37 as a condensation catalyst.

52. (Once Amended) Use of the fabric in accordance with [one of the Claims 37 to 44] Claim 37 as a floor covering.

53. (Once Amended) Use of the fabric in accordance with [one of the Claims 37 to 44] Claim 37 as a covering material.

REMARKS

The amendments to the specification as set forth above are intended to clarify and set apart the various sections of the subject application.

The amendments to the claims as set forth above are intended to remove all multiple dependent claims from the subject application and to more particularly point out and distinctly claim the subject invention.

Respectfully submitted,

JENKINS & WILSON, P.A.

Date: 2-3-00

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420 Rec'd PCT/PTO 03 FEB 2000

Process to manufacture a cellulose fibre from hydrate cellulose

The invention described here concerns a process to manufacture a cellulose fibre from hydrate cellulose, a cellulose fibre obtainable by this process, as well as a fabric which contains these cellulose fibres and the use of this fabric.

Absorbent fibrous materials which can also be applied for cleaning purposes are already known. Examples are cross-linked carboxy methyl cellulose (CMC), which can be manufactured in accordance with the process described in CH-A-491140, or viscose fibres, which contain hydrophilic polymer substances such as polyacrylic acid (BE-A-2324589), poly-N-vinyl pyrrolidone or CMC (DE-A-25 50 345), alginic acid (DE-A-27 50 622) or other copolymers (DE-A-27 50 900). Besides their high absorptive power, these fibres have good water-retention properties. The manufacture of these fibres, however, is associated with a high degree of technical complexity, and some of these fibres contain substances which either do not biodegrade at all or only with difficulty, so that natural disposal (e.g. composting) of the fibres subsequent to their use is not possible.

The object of this invention is to provide a process to permit the manufacture of a cellulose fibre from hydrate cellulose with an extremely large surface area and which biodegrades easily. Another object of the invention is to provide a fabric made from these fibres which is characterised by a high absorptive power, good water-retention properties, high grease-solvent properties as well as particle-absorbing properties, which is suitable for making products that are themselves easy to clean, which can be used for cleaning and decontamination as well as to reduce the surface tension of water and which can be disposed of without damage to the environment.

The above-described objects are solved by the invention-design process to manufacture a cellulose fibre from hydrate cellulose which comprises the following steps:

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Katrina A. Glenn

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- a) Treatment of wood pulp derived from shoots no older than 1 year of deciduous trees or conifers with an alkali metal hydroxide solution in order to obtain an alkali cellulose;
- b) pressing out of the superfluous alkali metal hydroxide solution from the obtained alkali cellulose;
- c) shredding of the alkali cellulose into crumbs;
- d) ripening of the alkali cellulose crumbs to a maturity of between 5° and 30° Hottenroth;
- e) application of the wet sulphide process to treat the ripened crumbs in order to sulphidise the cellulose;
- f) rinsing and diluting of the sulphidised cellulose with water in order to obtain a spinning solution;
- g) subsequent ripening of the rinsed and diluted cellulose to a maturity of between 5° and 30° Hottenroth;
- h) filtering and downstream deaeration of the spinning solution;
- i) injection of the spinning solution into a regenerating bath under application of spinnerets;
- j) stripping off the coagulating fibres with simultaneous twisting in order to obtain twisted fibres;
- k) dehydrating of the twisted fibres;
- l) desulphurisation of the twisted fibres;

m) washing of the twisted fibres with water;

n) predehydrating of the twisted fibres; and

o) drying of the twisted fibres;

and by a fabric comprising a backing fabric and a pile woven into the backing fabric containing these fibres.

Figures 1 to 6 show the microstructure of the invention-design fibres.

Figures 7 to 15 show the macrostructure of the invention-design fibres.

Figure 16 shows an example of the invention-design fabric.

Figures 17 to 20 show electron micrographs of bacteria which are adsorbed on the lamellae of the invention-design fibres.

Figure 21 shows a device to obtain water with a reduced surface tension.

Figure 22 shows a tensiometer to measure the surface tension.

Figures 23 to 28 show the surface tension-reducing effect of invention-design fabrics as a function of rinsing the fabric.

Figure 29 shows the history of the surface tension in the case of fabrics which remain in the water.

Figure 30 shows the surface tension-reducing effect of the invention-design fabric before and after a drying phase.

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Figure 31 shows an experimental set-up to measure the water-absorbing capacity of the invention-design fabric.

Figure 32 shows a model to permit calculation of the specific surface area of the invention-design fabric.

The invention-design process permits the manufacture of a biodegradable cellulose fibre from hydrate cellulose $(C_6H_{10}O_5)_n$, whose microstructure displays fibre-parallel lamellae. The preferred lamella spacing lies in the range between 1 nm and 5 μm , whereas the range between 200 nm and 1 μm is ideal. Another particularly good range is between 1 and 20 nm. As a result of this microstructure, the fibre has an extremely large surface area. In a preferred design, the invention-design fibre is birefractive. The microstructure and the macrostructure of the invention-design fibres were determined by means of the processes described below.

The microstructure of various invention-design fibres which were designated L1, L2, and S2 in the tests is shown in Figures 1 to 6. The purpose of the tests was to analyse the fibres as complete fibres with respect to the surface structure using a scanning electron microscope. The macrostructure of the fibres, shown in Figures 7 to 15, was analysed as a microtome section. The microtome sections were prepared by embedding the fibres in PMMA, cutting them and then extracting them from the embedding medium. The exposure of the fibres to high temperatures was thereby kept as low as possible.

Figures 1 to 6 show the resultant microstructures viewed from above. All invention-design fibres have a fibre-parallel lamellar structure. However, these figures are not able to give any indication of the cross-sectional structure (macrostructure). It is the microtome sections which show the cross-sectional structures, as shown in Figures 7 to 15. Fibre L1 was prepared using an oval spinneret, whereas an extended slit-shaped spinneret was used to prepare fibres L2 and S2. The qualitative results of the tests are summarised in the following table (Table 1).

TABLE 1

Fibre type	L1	L2	S2
Cross-sectional form (macrostructure)	globular, strongly fissured	lamellar, fissured	lamellar, fissured
Spec. surface area (qualitative)	extremely large	medium	large
Position of surface structure (incisions)	on all sides	on all sides	mainly on one side, i.e. on the inside after curling
Max. fibre width [μm]	approx. 35	approx. 80	approx. 200
Curling effect after cutting (indication of internal stress)	not detectable	low	high

Wood pulp derived from shoots no older than 1 year of deciduous trees or conifers was used to prepare the invention-design fibre. It is particularly easy to remove the lignin from such a base material. Ideally, wood pulp derived from shoots no older than 1 year of false acacia trees, teak trees, bongassi trees or bamboo is used, although wood pulp derived from shoots no older than 1 year of comparable European trees can also be used. The lignin content of the less-than-one-year-old shoots used should be as low as possible and is preferably no more than 7%. In a particularly preferred invention design, the lignin content of the base material is no more than 5%, and ideal is no more than 2%.

This wood pulp is treated with an alkali metal hydroxide solution, preferably at a temperature of between 15 and 25°C, in order to obtain an alkali cellulose. It is preferable to use a sodium hydroxide solution which contains between 150 and 350 g/l of sodium hydroxide as the alkali metal hydroxide solution. A sodium hydroxide content of approx. 300 g/l is particularly favourable.

The superfluous alkali metal hydroxide solution is then pressed out of the resultant alkali cellulose, e.g. under application of a submersible press.

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The alkali cellulose is then shredded into crumbs, whereby shredding can include a coarse comminution step (e.g. in a pre-shredder) and a fine comminution step (e.g. in a disc mill).

The crumbs are then fed, for example, to a maturing drum and ripened to a maturity of between 5° and 30° Hottenroth, preferred is a maturity of between 8° and 12° Hottenroth, and a maturity of approx. 10° Hottenroth is ideal. The preferred temperature during the ripening process is between 60 and 80°C, particularly favourable is a temperature of between 65 and 75°C, and approx. 72°C is absolutely ideal. The ripening process can then be slowed down by reducing the temperature to between 40 and 50°C, or preferably to approx. 45°C.

The ripened crumbs are subsequently treated under application of the conventional wet sulphide process in order to sulphidise the cellulose. The wet sulphide process is preferably carried out in a solution containing carbon disulphide, sodium hydroxide and Berol. The preferred carbon disulphide content of the solution is between 150 and 250 g/l, particularly favourable is between 180 and 210 g/l, and the preferred content of sodium hydroxide is between 250 and 350 g/l, particularly favourable is between 280 and 320 g/l, and the preferred content of Berol is between 100 and 200 g/l, particularly favourable is approx. 150 g/l. The most preferable type of Berol used for this process step is one of the commercially available products from Berol-Kemie Ltd., 44401 Stennungsund, Sweden.

After sulphidisation, the sulphidised cellulose is rinsed and then diluted with water to produce a spinning solution. The cellulose is then subsequently ripened to a maturity of between 5° and 30° Hottenroth, whereby a maturity of between 8° and 12° Hottenroth is preferred. The degree of ripeness achieved during initial ripening is diminished by the sulphidisation process and by rinsing and diluting the sulphidised cellulose, and it is not until a subsequent ripening process is carried out that the desired degree of ripeness is finally achieved. In practical operation, it is not always easy to control the degree of ripeness with great accuracy. In this case, two or more

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batches of spinning solutions can be mixed in order to achieve the desired degree of ripeness.

The downstream filtration of the spinning solution may be carried out under application of filter presses. The spinning solution is then deaerated.

The deaerated spinning solution is introduced by means of spinnerets into a regenerating bath, preferably at a temperature of between 35 and 45°C, and ideally at a temperature of approx. 40°C. A suitable regenerating bath contains between 70 and 160 g/l of sulphuric acid, preferred is between 90 and 140 g/l, and approx. 120 g/l is ideal, plus between 0.3 and 4 g/l of zinc sulphate, preferred is between 0.5 and 2 g/l, and approx. 1 g/l is ideal, plus between 0.05 and 1 g/l of Berol, preferred is between 0.1 and 0.7 g/l, and approx. 0.4 g/l is ideal. The most preferable type of Berol used for this process step is one of the commercially available products from Berol-Kemie Ltd., 44401 Stennungsund, Sweden. The spinnerets used can be oval to long-slit-shaped, and are heated to keep them within a preferred temperature range of 55 - 75°C, particularly favourable is between 65 and 70°C, and approx. 67°C is absolutely ideal.

The fibres are stripped off as they coagulate and simultaneously twisted in order to obtain twisted fibres, which are then dehydrated. A sulphuric acid solution, for example, can be used for dehydrating, whereby a content of ≤ 15 g/l of sulphuric acid is preferred and ≤ 10 g/l is ideal.

Desulphurisation of the twisted fibres is generally carried out in a sodium sulphate solution, which preferably contains between 2 and 5 g/l of sodium sulphate and ideally approx. 3 g/l. Other desulphurisation processes are also possible. The fibres are then washed with water.

After washing, the fibres can be further treated, for example in order to modify the optical properties of the fibres. Titanium dioxide, for example, can be used to give the fibres a dull finish.

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The fibres are then predehydrated and dried, whereby if the lamellar structure of the fibres is to remain intact, as little mechanical stress as possible must be applied to the fibres during predehydrating. Predehydrating can take place under application of compressed air, for example, and drying under application of tunnel dryers, for example, although other suitable processes and equipment known to the specialist can also be employed.

Under application of the invention-design process, a fibre is yielded which contains practically no more lignin and which is substantially free from sulphuric acid and carbon disulphide. Because of its lamellar microstructure, this fibre has an extremely large surface area. It is impossible to obtain such a large surface area with conventional fibres made from sulphite cellulose, because the sulphite pulping process leads to destruction of the lamellar structure.

The fibre produced in this manner has a preferred count of 330 dtex or more.

The fibre produced as described above can be used to manufacture a fabric which is characterised by a high absorptive capacity, good liquid-retention properties, high grease-solvent properties as well as particle-absorbing properties.

The invention-design fabric comprises a backing fabric and a pile woven into the backing fabric which contains the fibres manufactured in the manner described above.

The backing fabric preferably has a lattice-like structure. The backing fabric and pile can consist of the same type of fibre, although this is not essential. Durability criteria can make it necessary to use stronger backing fabric fibres, for example. The backing fabric preferably contains some viscose staple fibres; a backing fabric which consists exclusively of such fibres is ideal.

In a preferred invention design, the pile forms a fibre bed of approx. 0.5 cm in height above the backing fabric. The pile should preferably contain oval or tape fibres or a mixture of the two. A fabric whose pile contains a lot of oval fibres but only a small amount of tape fibres has especially high grease-adsorptive properties. A fabric whose pile contains a small amount of oval fibres but a lot of tape fibres is particularly suitable for reducing the surface tension of water.

In another preferred invention design, the pile consists of 50% oval and 50% tape fibres. And in a particularly preferred invention design, the pile consists of 50% of oval fibres with a count of 330 dtex F60 and 50% of tape fibres with a count of 330 dtex F80. Such a fabric is shown in Figure 16.

The invention-design fabric is characterised especially by the following properties:

- it can bind bacteria and particles of dirt reversibly and can then be cleaned without the use of chemicals;
- it has an extremely large specific surface area;
- it has high specific fluid-retention properties; and
- it can be disposed of with minimum environmental impact.

The above-described properties give rise to a wide range of applications for the invention-design fabric.

Because the invention-design fabric is pH neutral, it can, for example, be used to manufacture personal hygiene articles which are kind to the skin, e.g. sanitary pads, as well as wash cloths to clean the body and skin, and especially to clean mucous membrane (stoma cleaning), and also to clean the skin if skin infections and neurodermatitis are a problem or as a wash cloth on the sector of personal hygiene for the aged and infirm. When using the fabric as a wash cloth, the skin surface can

be cleaned of water-soluble and greasy cosmetics in an allergy-free and pH-neutral manner without the need of special cleansing agents. The fabric itself can then be cleaned of bacteria and cosmetics by simple mechanical cleaning (rinsing and wringing out) in cold water. This aspect gains particular significance if the skin is sensitive or damaged, e.g. as is the case with neurodermatitis or acne. Use of the invention-design fabric with nothing else but water eliminates the possibility of additional skin irritation or damage. Germs, for example, can thus be removed from the skin more gently than with detergent surfactants or disinfectants.

The invention-design fabric can also be used as an udder cloth to clean and decontaminate cow's udders before connection to a milking machine. This rules out the possibility of germs being introduced into the milk.

On the other hand, the invention-design fabric can also be used to manufacture products which are themselves easy to clean, e.g. textiles and fabric for clothing; such products can then be cleaned without the need for any chemicals even if strongly soiled by foodstuff residues, etc. Examples of such products are bed linen, table linen, work clothes or baby articles (e.g. nappies, bibs and wash cloths), upholstery coverings for furniture or car seats as well as covering fabrics for stuffed toys. Stains left by ketchup, juice, red wine, lipstick or blood, to name just a few examples, can be removed from these products without residue by simple rinsing in cold water. Products such as nappies, wash cloths or bibs can also be washed residue-free without the need for washing detergents at temperatures of up to 40°C. It is also possible to wash the products in a washing machine either on a cold cycle or one up to 40°C without the need for any washing detergents. The products can then be simply hung up to dry or can be dried in a dryer (up to 40°C).

Another application for the invention-design fabric is the employment as a floor covering for special-purpose rooms (e.g. humid rooms) or hygienically sensitive rooms.

The invention-design fabric can also be used as a condensation catalyst to condense steam or humidity, e.g. as a "roof panel" for shower or bath cabins or humid rooms. The moisture is absorbed by the covering and then - during the course of a slow drying process - is released into the atmosphere again in a retarded manner. This prevents the rapid condensation of steam or moisture on the room walls, even if the room in question is badly ventilated (e.g. as in old buildings). The covering made from the invention-design fabric remains free from fungi, bacteria and algae.

Another possible application for the invention-design fabric is the use as a particle filter, e.g. to remove particles or micro-organisms from organic and inorganic fluids. To this end, the fabric can be layered in parallel layers, for example, or rolled.

The biophysical properties of the invention-design fabric are a result of the lamellar microstructure of the fibres on the one hand, and of the configuration of the fibres on the surface of the fabric on the other hand.

The following is a description of the employment of the invention-design fabric for cleaning and decontamination. This application permits organic surfaces (e.g. skin) or inorganic surfaces (e.g. objects, floors and windows) to be cleaned and the bacteria present on these surfaces to be removed without the need for disinfectants, meaning that the surfaces being cleaned are subject to neither chemical nor thermal stress. In contrast to conventional decontamination with disinfectants, decontamination with the invention-design fabric achieves the same or better germicidal effects without any selective processes caused by resistance to chemical attack occurring.

The invention-design fabric is wetted with water so that a certain residual moisture content (e.g. approx. 20%) remains. At this degree of residual moisture, the surface to be decontaminated can be mechanically decontaminated, whereby the ability of the fabric to reduce the surface tension of water leads to an improved lipid solubility. With a fabric measuring approx. 600 cm² in surface area, a highly contaminated

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surface of 1 m² can be cleaned optimally. The fabric itself is subsequently decontaminated by being steeped in water and moved to and fro mechanically. This type of cleaning serves to completely decontaminate the fabric, whereby every single contaminated particle is released into the water. The fabric itself remains biologically and chemically inert during this process. In the case of a surface to be cleaned of approx. 30 m², a contamination saturation of the cleaning water is reached at about 10 litres of water, although this is dependent on the degree of contamination of the surface. The adsorptive effect which exists when the fabric contains approx. 20% residual moisture is preserved for future cleaning procedures provided that no impermissible chemical, mechanical or thermal stresses destroy the fibre structure. The optimum temperature range for use of the fabric lies between 5 and 30°C. The adsorptive effect is destroyed as soon as the temperature exceeds 60°C. Intensive contact (impregnation) with detergents destroys the function of the fabric because it damages the surface structure. Contact with 0.1 standard acids or alkaline solutions or with alcoholic solutions presents no problem.

Figures 17 to 20 show electron micrographs (magnified 2,000, 5,000, 20,000 and 30,000 times) of bacteria (here: staphylococci) which are adsorbed on the lamellae of the invention-design fibres.

Surprisingly, it turned out that the invention-design fabric was also capable of reducing the surface tension of water by as much as 20% or more without the use of any chemicals. Water of this nature with a reduced surface tension can be used for extraction processes or synthesis as well as for fermentation processes - for example in brewing operations.

To achieve a reduction of the surface tension, the water is brought into contact with the invention-design fabric. The effect manifests itself within a short time, is practically independent of the water temperature and lasts for about two hours.

Figure 21 shows a device to obtain water with a reduced surface tension. The device comprises, e.g. a vessel filled with water which is equipped with an overflow and an outflow. Several invention-design fabrics can be put into the vessel simultaneously.

The following examples and the figures illustrate the invention.

Example 1: Fabric for cleaning contaminated surfaces

Test-rig configuration

1.1 Tested bacterial strains

- E. coli
- Staphylococcus aureus
- Streptococcus pyogenes
- Enterococci (S. faecium)
- Streptococcus bovis
- Pseudomonas pyocyanea

1.2 Cultures

Cultures prepared by means of incubation in nutrient broth or litmus milk (mixture of litmus and milk) or in nutrient agar at between 25 and 30°C until the bacterial count reaches $> 10^8$ bacteria/ml. The number of bacteria was determined (corresponds to the primary bacterial count = 100%).

1.3 Test procedure

The fabric was immersed in the above-described medium with a bacterial concentration $> 10^8$ bacteria/ml for a period of 15 minutes. The fabric was then placed (not rinsed) in lukewarm water (10 - 20°C) for between 1 and 3 minutes. It was then removed from the water and put into a sterile nutrient solution, which was

then incubated. The bacterial concentration in the nutrient solution was determined after a period of between 24 and 72 hours. The measured values were related to the primary bacterial count, whereby the following results were achieved:

	I	II	III
E. coli	15%	12%	9%
Staph. aureus	18%	16%	16%
Strept. pyogenes	5%	6%	4%
Enterococci	7%	5%	8%
Strept. bovis	17%	12%	14%
Pseudom. pyogenes	6%	4%	3%

Subsequent to re-incubation, the measured bacterial densities/concentrations of all bacterial strains reduced markedly.

Example 2: Germicidal properties of the invention-design fabric used to clean the udders of dairy cows

The tests were carried out on 5 cows each in 3 dairy farms during 10 milking periods. One half of the cows' udders was cleaned with the cloth the farm normally used and then soaked in disinfectant, whereas the other half was cleaned with the invention-design fabric. The udder halves were changed at every milking time. After cleaning, the invention-design fabric was placed in water with no detergents or disinfectants. The comparison cloths were also placed in water, whereby on one farm, a disinfectant was added to the water. Before and after the udder cleaning, an impression culture was prepared on ass. agar (antibiotic sulphonamide sensitivity agar) from each cloth and was incubated for 18 hours at between 38 and 40°C.

Results:

In differentiating the bacteria by means of selective cultures, it was predominantly the following bacteria which were found:

- E. coli
- Streptococci
- Staphylococci
- Enterococci

Assuming the primary bacterial count to be 100%, we arrived at the following values:

	Invention-design fabric	Farm's own cleaning cloth	
		without disinfectant	with disinfectant
E. coli	10%	92%	35%
Streptococci	3%	87%	25%
Staphylococci	2%	78%	20%
Enterococci	9%	83%	41%

The statistical evaluation of the results clearly shows the germicidal property of the invention-design fabric. In other words, the risk of transferring germs from one cow to another during milking is significantly reduced with the invention-design fabric.

Example 3: Cleaning a contaminated invention-design fabric - comparison with a conventional cotton fabric

The invention-design fabric was cut into squares measuring 6 x 6 cm which were then used to wipe contaminated surfaces. The comparison fabric - a cotton fabric which had been washed several times - was also cut into pieces of about the same weight (see Tables 2 - 6). The test surfaces consisted of glazed ceramic tiles (5 x 5 cm) which had been inoculated with one of the test bacterial strains, i.e. either *Enterococcus faecium*, *Escherichia coli* or *Staphylococcus aureus*. The tiles were each inoculated with 50 µl of an overnight culture of the test bacteria, so that the number of bacteria to be recovered from the tiles was between 3.4×10^5 and 8.9×10^6 . The contamination degree of the test surfaces was controlled in two ways: the bacterial count was determined in the inoculation suspension; and the inoculated surfaces were rinsed, dried and then analysed. Drying took place by means of a 20-minute exposure at approx. 25°C in a laminar flow workbench. The surfaces were

then wiped with the test fabrics and the bacterial count on the tiles was determined by rinsing the tiles in 100 ml of sterile, distilled water and by distributing some of the rinsing solution on nutrient agar slides with a spatula. The number of bacteria taken up from the tiles into the test fabrics was also determined by rinsing at room temperature in 200 ml of distilled water, this process being assisted mechanically by manually wringing the fabrics out, and the rinsing solution was then inoculated onto nutrient agar slides. The number of bacteria remaining in the test fabrics after rinsing was determined by means of a second rinse, and in the last test series by means of a third rinse. The following tables therefore show the bacterial counts of the following suspensions:

- Overnight culture (inoculation suspension)
- Rinsing water used to rinse the inoculated surface
- Rinsing water used to wipe the surface
- Rinsing water from the fabric after wiping the tiles
- Rinsing water from the fabric after the first rinse
- Rinsing water from the fabric after the second rinse

The test results are summarised in the following tables. Table 2: *E. coli* ATCC 11229; Table 3: *Enterococcus faecium*; Table 4: *Enterococcus faecium*; Table 5: *Staphylococcus aureus* ATCC 6538; and Table 6: tests with all three test bacteria. The test parameters applied for Tables 2 to 5 were more or less identical. The difference between Tables 3 and 4 (both with *Enterococcus faecium*) is the lower localised propagation on the inoculated surfaces. In Table 6, both the test fabric and the control fabric were rinsed once again to determine whether this made any difference to the bacterial count in the fabric over that of a fabric which had been rinsed only once.

TABLE 2

Bacteria: *E. coli* ATCC 11229

Inoculation with overnight culture KBE/50 μ l	I		II		III	
	Invention- design fabric	Cotton	Invention- design fabric	Cotton	Invention- design fabric	Cotton
- on inoculated surface	5.6×10^5	5.6×10^5	3.7×10^5	3.7×10^5	3.4×10^5	3.4×10^5
- in fabric after wiping surface	4.8×10^5	4.0×10^5	3.8×10^5	3.0×10^5	5.8×10^5	1.8×10^5
- in fabric after rinsing	7.0×10^3	6.0×10^3	3.0×10^3	6.0×10^3	5.0×10^3	4.0×10^3
- on tile after wiping	1.2×10^4	2.3×10^4	0.0	6.5×10^3	3.6×10^4	6.0×10^3
Weight 6 x 6 cm in g	1.941		1.998	0.875	2.106	1.951

TABLE 3

Bacteria: Enterococcus faecium

Inoculation with overnight culture KBE/50 µl Measured bacterial count	I 2.0 x 10 ⁷ on tile		II 2.1 x 10 ⁷ on tile		
	Invention- design fabric	Cotton	Invention- design fabric	Invention- design fabric	Cotton
- on inoculated surface	4.8 x 10 ⁵	4.8 x 10 ⁵	6.0 x 10 ⁵	6.0 x 10 ⁵	6.0 x 10 ⁵
- in fabric after wiping surface	3.6 x 10 ⁵	3.2 x 10 ⁵	5.0 x 10 ⁵	4.0 x 10 ⁵	1.8 x 10 ⁵
- in fabric after rinsing	2.0 x 10 ³	5.0 x 10 ³	4.0 x 10 ³	3.0 x 10 ³	2.0 x 10 ³
- on tile after wiping	3.0 x 10 ³	1.0 x 10 ⁴	5.0 x 10 ³	4.0 x 10 ³	3.0 x 10 ³
Weight 6 x 6 cm in g	1.679	1.017	1.852	1.784	0.950
Initial germination of cloth	1.0 x 10 ³		3.0 x 10 ³	3.0 x 10 ³	

TABLE 4

Bacteria: *Enterococcus faecium*

Inoculation with overnight culture KBE/50 µl Measured bacterial count	III 1.8 x 10 ⁷ on tile		IV 3.0 x 10 ⁷ on tile	
	Invention- design fabric	Invention- design fabric	Invention- design fabric	Cotton
- on inoculated surface	8.9 x 10 ⁶	4.9 x 10 ⁵	8.9 x 10 ⁶	6.7 x 10 ⁵
- in fabric after wiping surface	6.2 x 10 ⁸	2.6 x 10 ⁵	4.8 x 10 ⁵	4.6 x 10 ⁵
- in fabric after rinsing	7.9 x 10 ⁴	5.0 x 10 ³	3.6 x 10 ⁴	6.0 x 10 ³
- on tile after wiping	7.1 x 10 ⁴	5.0 x 10 ³	5.6 x 10 ⁴	5.5 x 10 ³
Weight 6 x 6 cm in g	1.948	1.765	0.775	1.968
Initial germination of cloth	2.0 x 10 ³	4.0 x 10 ³		

TABLE 5

Bacteria: *Staphylococcus aureus* ATCC 6538

Inoculation with overnight culture KBE/50 μ l	I Staph. aureus 7.5×10^7 on tile		II Staph. aureus 4.0×10^7 on tile		III Staph. aureus 6.0×10^7 on tile	
	Invention- design fabric.	Cotton	Invention- design fabric.	Cotton	Invention- design fabric	Cotton
Measured bacterial count						
- on inoculated surface	4.8×10^6	4.8×10^6	2.3×10^6	2.3×10^6	7.0×10^6	7.0×10^6
- in fabric after wiping surface	4.6×10^6	1.1×10^6	1.6×10^6	1.0×10^5	7.0×10^6	5.0×10^6
- in fabric after rinsing	6.6×10^4	5.0×10^2	1.6×10^4	0.0	9.0×10^4	1.3×10^5
- on tile after wiping	1.9×10^5	6.0×10^4	6.4×10^4	1.4×10^4	7.4×10^4	7.5×10^4
Weight 6×6 cm in g	1.987		1.855	0.960	1.939	1.937

TABLE 6

Inoculation with overnight culture KBE/50 μ l Measured bacterial count	E. faecium 3.9×10^7 on tile		E. coli 3.8×10^7 on tile		Staph. aureus 6.0×10^7 on tile	
	Invention- design fabric	Cotton	Invention- design fabric	Cotton	Invention- design fabric	Cotton
- on inoculated surface	1.0×10^6	1.0×10^6	3.8×10^5	3.8×10^5	6.8×10^6	6.8×10^6
- in fabric after wiping surface	4.6×10^5	5.0×10^5	3.8×10^5	4.4×10^5	5.0×10^6	2.6×10^6
- in fabric after rinsing once	8.0×10^3	1.0×10^4	7.2×10^3	8.8×10^3	4.6×10^4	1.4×10^5
- on tile after wiping	6.0×10^2	3.3×10^3	1.5×10^3	5.2×10^3	7.8×10^4	1.0×10^5
- in fabric after rinsing twice	6.0×10^1	8.4×10^2	2.0×10^2	6.8×10^2	6.0×10^2	4.4×10^3
Weight 6 x 6 cm in g	1.933	2.0	2.034	2.0	2.015	2.0

The quantitative evaluation of the test results shows that the tiles are cleaned to more or less the same degree by both the invention-design fabric and the cotton fabric. Although there are only signs that the invention-design fabric has a better cleaning effect, a distinct difference can be seen in the measured bacterial count in the fabrics after use. At this point, a greater number of the micro-organisms remain in the invention-design fabric than in the cotton fabric. After the first rinse, the bacterial counts in both fabrics are more or less the same, but after the second rinse (Table 6), the bacterial count in the invention-design fabric is below that of the cotton fabric in all three analyses.

The results show that the invention-design fabric can be freed of the absorbed bacteria by simple rinsing in pure water better than the conventional cotton fabric, and that it is superior to the cotton fabric when it comes to cleaning the surfaces. The invention-design fabric is therefore especially suitable for cleaning surfaces and for other applications where a reversible absorption of bacteria is required.

Example 4: Preparation of water with a reduced surface tension

To demonstrate the preparation of water with a reduced surface tension, fresh tap water with a hardness degree of between 5 and 25 and any mixture of ions is preferred; the process can be carried out within a preferred temperature range of between 5 and 30°C, or ideally between 15 and 25°C, and leads to water with a distinctly reduced surface tension. It is preferable to use a container with a completely smooth surface, e.g. of glass, metal, enamel or ceramic, in which the water can make contact with the invention-design fabric.

The invention-design fibre should preferably be woven into a double-sided fabric with a bed of fibres on each side which are 0.5 cm in length. Assuming this and the fact that the cloths are spaced in water at uniform intervals, a surface area of 1 m² of fabric with double-sided pile for 0.16 m³ of water at a residence time of between 5 and 10 seconds is required for an optimal degree of surface-tension reduction. The water vessel used can be any shape. The fabric can be clamped in place in a basin

or if preferred, suitable mechanical means can be employed to dip the fabric into a basin. After the fabric is removed from the vessel or the water is removed from the vessel, the water retains its reduced surface tension at temperatures of up to 40°C for at least 60 minutes, whereas the surface tension gradually rises again to normal values (72 to 78 mN/m) after 120 minutes. During this time, the water can be integrated into synthesis, extraction or fermentation processes, both with or without fibre contact. Whereby it is also possible, for example, to introduce the water at all fermentation stages - even at temperatures of above 40°C - in order to improve and accelerate the fermentation processes during brewing.

In the same way, it is also possible after the mash has cooled and before the yeast has been added to reduce the surface tension by means of the dipping method in order to achieve better brewing results.

Example 4.1:

In this example, 3 invention-design fabric samples "S10", "L01", and "L02" were investigated, samples which differ in terms of their surface structure quality.

The measurements were made in glass vessels, whereby an effective surface area of the fabric samples measuring 400 cm² was analysed in 4 litres of water. The temperature during the tests was around 20°C.

The measurements were carried out

1. without previous rinsing
2. after three rinses
3. after six rinses
4. after nine rinses

of the respective fabric sample.

The comparison measurements were carried out in bidistilled water and in fresh water taken from the tap.

The surface tension was measured with the tensiometer shown in Figure 22.

Test description:

A 5-litre vessel of Duran glass is filled with 4 litres of fresh tap water of 20°C, and a sample of this water is extracted to measure the surface tension. The test fabric is then immersed in the water, kneaded a few times and then removed after being wrung out. Several samples are taken of the water remaining in the glass vessel, and the surface tension is measured. In the same way as described above, the fabric is now rinsed twice without subsequent measurement in two lots of fresh water measuring 4 litres each. The entire process is repeated three times.

The surface tension is measured as follows: The water sample is filled into the sample dish of the tensiometer and the dish is then placed on the lifting table and raised until the platinum ring immerses in the sample. The servomotor is then activated which lowers the water sample on the table, whereby a water leaf at the ring is extracted. The motor stops at maximum stress as soon as the surface film starts to give and there is no more tensile load acting on the balance from which the ring is suspended. The maximum value can be read off the digital display.

One-sided fabric:

First of all, the effect of one-sided fabric samples of 400 cm² in size is investigated. To this end, each fabric sample is placed in 4 litres of fresh water of 20°C and removed after being stirred several times in the water. This takes place initially with unrinsed fabrics. A sample is then extracted and the surface tension measured.

The fabric is then rinsed three times and the test repeated. The test is also repeated after each fabric has been rinsed six times and nine times.

Figures 23 to 25 show the surface tension values measured after zero, three, six and nine rinses plotted against those of bidistilled water and fresh water. In the case of all rinses, a high introduction of air into the test water was observed, as well as "fuzzing", which caused the measured values to fluctuate. Because of this, several measurements were made and the arithmetic mean shown in each case.

In the case of Figures 23 to 25, the same statement in broad terms can be made: Immersing the respective fabric in the test water reduces the surface tension by 30-40%, from approx. 71 mN/m to 40-50 mN/m. The surface tension-reducing effect of the respective fabric decreases in proportion to the number of times the fabric is rinsed.

Double-sided fabric:

In comparison to the single-sided fabrics, the double-sided fabrics display more than double the mass at the same surface area of 400 cm². The measured values of each fabric are shown in Table 7.

TABLE 7: Mass of tested fabric

Type	One-sided	Double-sided
L01	16.10 g	38.87 g
L02	16.69 g	38.44 g
S10	15.53 g	41.31 g

The surface tension-reducing effect of the double-sided fabric as a function of the number of rinses is shown in Figures 26 to 28.

The double-sided fabrics L01 and S10 reduce the surface tension of the sample from around 72 mN/m to 60-62 mN/m. This corresponds to a reduction of 14-17%. This effect is independent of the number of rinses and remains on the same level even after 9 rinses.

Initially, the double-sided fabric L02 succeeds in reducing the surface tension by 42%, from 72 mN/m to 42 mN/m. However, the surface tension-reducing effect decreases as the number of rinses increases, and final measurement after 9 rinses showed a value of 70.4 mN/m.

Surface tension when fabrics remain in the water:

In this test, the respective fabrics were not removed from the water before measuring the surface tension. Several measurements were made one after the other in rapid succession. Figure 29 shows the surface tension gradient of fabrics L01, L02, and S10 as a function of the time. A constant value did not set in until after about 10 minutes. The water temperature was a constant 20°C.

Whereas the value of the water with the fabrics L01 and L02 set in at between 69 and 68 mN/m, the water with the fabric S10 displays a distinctly lower final value of 62 mN/m.

In summarising, we can state that:

The first contact of the invention-design fabric with water succeeded in reducing the surface tension of fresh water by up to 40%, from approx. 72 mN/m to values around 40 mN/m. This effect, however, diminishes in the case of the one-sided fabrics in proportion to the number of rinses, as the repeated tests showed. In the case of the double-sided fabrics L01 and S10, these fabrics were each still able to reduce the surface tension of fresh water by 14-17% even after being rinsed nine times. A constant value set in here which differed only negligibly from the values measured after 3 and 6 rinses.

The double-sided fabric L02 behaved in an analogous way to the one-sided fabrics, where the surface tension-reducing effect diminished with increasing number of rinses.

If the fabrics are left in the water, a constant surface tension value of 62 mN/m sets in after about 10 minutes under ideal conditions. In comparison with fresh water (72 mN/m), this means a reduction of the surface tension by 14%.

Example 4.2:

In this test, the surface tension-reducing effect on water of the double-sided fabric L01 described in Example 4.1 after a drying phase of several months was tested.

The measurements were carried out in glass vessels. An effective surface area of 400 cm² of fabric was investigated in 4 litres of water. The temperature was 20°C.

The comparison measurements were carried out in bidistilled water and fresh tap water from the mains.

The surface tension was measured with the tensiometer shown in Figure 22.

Test description:

A 5-litre vessel of Duran glass is filled with 4 litres of fresh tap water, and one with bidistilled water, whereby the temperature was 20°C in each case, and a sample of water is extracted from each vessel to measure the surface tension. The test fabric is then immersed, kneaded a few times and removed after being wrung out. Several samples are taken of the water remaining in the glass vessels, and the surface tension is measured.

The surface tension is measured as follows: The water sample is filled into the sample dish of the tensiometer and the dish is then placed on the lifting table and raised until the platinum ring immerses in the sample. The servomotor is then activated which lowers the water sample on the table, whereby a water leaf at the ring is extracted. The motor stops at maximum stress as soon as the surface film starts to give and there is no more tensile load acting on the balance from which the ring is suspended. The maximum value can be read off the digital display.

Figure 30 compares the surface tension of water after contact with fabric "L01" investigated both before and after the drying phase, as well as after contact with fresh tap water and bidistilled water. During measurement, it was registered that a large amount of air was introduced into the test water, which led to fluctuating values. Because of this, several measurements were made and the arithmetic mean shown in each case. The individual measurements are shown in Table 8.

TABLE 8: Surface tension-reducing effect of the invention-design fabric "L01" before and after the drying phase (individual values).

Single measurement	Surface tension (mN/m)	
	L01 prior to drying	L01 after drying
1	62.0	58.2
2	62.4	58.4
3	62.5	58.3
4	62.1	58.4
5	62.5	58.7
Mean value x	62.3	58.4

The surface tension-reducing effect of the double-sided fabric "L01" did not deteriorate after the drying phase. It was still capable of reducing the surface tension from 71.4 mN/m to 58.4 mN/m, i.e. by 18%.

This test shows that the surface tension-reducing effect of the invention-design fabric can be attributed to the fibre structure, and not to substances leached from the fabric. The surface tension-reducing effect is therefore retained even after a prolonged period of drying.

Example 4.3:

Tables 9 and 10 show results from other tests which were carried out. The fabrics were all rinsed once or twice with water before the test, in order to remove any impurities left over from the manufacturing process.

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TABLE 9: Measuring results with different vessels

Measurement No.	Vessel	Amount [litres]	Temperature [°C]	Comments	Waiting time [min]	Surface tension before treatment [mN/m]	Surface tension after treatment [mN/m]
1	Glass	4	17	bidistilled water	0	71.9/71.9	-
2	Glass	4	17	tap water (TW)	0	71.7/71.6	-
3	Glass	4	16	TW + fabric (rinsed once)	0	71.6	52.4
4	Glass	4	15	TW + fabric (rinsed twice)	0	72.2	52.5
5	Plastic	10	14	TW + fabric (rinsed twice)	0	-	65.4
6	Glass	4	13	TW + fabric (rinsed twice)	0	-	58.8
7	Plastic	4	14	TW + fabric (rinsed twice)	0	72.6	62.2
8	Plastic	10	14	TW + fabric (rinsed twice)	0	71.1	60.7

TABLE 10: Measuring results at different temperatures

1	Glass	4	15	TW + fabric	0	69.7	59.9
2	Glass	4	20	TW + fabric	0	68.7	48.0
3	Glass	4	25	TW + fabric	0	66.4	46.5
4	Glass	4	30	TW + fabric	0	65.5	46.5
5	Glass	4	35	TW + fabric	0	64.4	46.4

Determination of the water-absorbing capacity of the invention-design fabric

The weight-related water-absorbing capacity or retention capacity of the invention-design fabric was investigated. The water-retention capacity of the fabric was investigated on a surface inclined by 45° for 60 seconds at ambient temperature and water saturation. The arrangement as shown in Figure 31 was employed.

Test description:

The dry fabric was first weighed and then soaked in a sufficient quantity of water for 20 minutes. The dripping-wet fabric was subsequently placed on an inclined plane (Figure 31) for 60 seconds and then weighed again immediately. The water-absorbing capacity is derived from the difference between the weight of the waterlogged fabric and the dry weight of the fabric. This test was repeated about 8 times, whereby the fabric was returned to the water for 2 minutes between each test to absorb water. The arithmetic mean was determined from the individual values. The test was carried out at ambient temperature (approx. 22°C).

Table 11 shows the water-retention capacity of the fabrics. The fabrics were each soaked for 20 minutes before the tests started. There was an interval of 2 minutes between each measurement.

TABLE 11: Water-retention capacity of the fabrics

Fabric	Dry weight	Drained weight	Water mass	Water-retention capacity (x times dry weight)
	[g]	[g]	[g]	
L01 single-sided	16.1	84.04	67.94	4.22
L02 single-sided	16.69	110.04	93.35	5.59
S10 single-sided	15.53	99.72	84.19	5.42
L01 double-sided	35.87	188.08	152.21	4.24
L02 double-sided	38.44	267.81	229.37	5.97
S10 double-sided	41.31	207.88	166.57	4.03

The amount of water retained by the respective fabrics was determined from the difference between the drained and the dry weight. Table 11 shows that each fabric is capable of retaining between 4 and 6 times its dry weight in water.

Model to permit calculation of the specific surface area of the invention-design fabric

The following values were assumed as examples for this calculation (see Figure 32):

Filament width	80 μm
Filament base structure height	4 μm
Lamella height	2 μm
Lamella width	1 μm

It therefore follows that:

- 1.) Cross-sectional line length of a filament
 $2 \times 40 \times 6 \mu\text{m} + 2 \times 4 \mu\text{m} = 488 \mu\text{m}$
- 2.) Production-specific filament length: 0.5 cm

Surface area of a single filament:

$$488 \mu\text{m} \times 0.5 \mu\text{m} = 244 \times 10^{-4} \text{ cm}^2$$

One fibre contains 80 filaments; the average number of fibres per square millimetre is 9 fibres.

This equates to a specific surface area per square centimetre of $72000 \times 244 \times 10^{-4} \text{ cm}^2 = 1756.8 \text{ cm}^2$ surface area per square centimetre of fabric area.

A cloth made of double-sided fabric measuring 20 cm x 23 cm (460 cm^2) therefore has a surface area of 161.62 m^2 .

Patent Claims

1. Process to manufacture a cellulose fibre from hydrate cellulose which comprises the following steps:
 - a) Treatment of wood pulp derived from shoots no older than 1 year of deciduous trees or conifers with an alkali metal hydroxide solution in order to obtain an alkali cellulose;
 - b) pressing out of the superfluous alkali metal hydroxide solution from the obtained alkali cellulose;
 - c) shredding of the alkali cellulose into crumbs;
 - d) ripening of the alkali cellulose crumbs to a maturity of between 5° and 30° Hottenroth;
 - e) application of the wet sulphide process to treat the ripened crumbs in order to sulphidise the cellulose;
 - f) rinsing and diluting of the sulphidised cellulose with water in order to obtain a spinning solution;
 - g) subsequent ripening of the rinsed and diluted cellulose to a maturity of between 5° and 30° Hottenroth;
 - h) filtering and downstream deaeration of the spinning solution;
 - i) injection of the spinning solution into a regenerating bath under application of spinnerets;
 - j) stripping off the coagulating fibres with simultaneous twisting in order to obtain twisted fibres;

- k) dehydrating of the twisted fibres;
 - l) desulphurisation of the twisted fibres;
 - m) washing of the twisted fibres with water;
 - n) predehydrating of the twisted fibres; and
 - o) drying of the twisted fibres.
2. Process in accordance with Claim 1, **characterised in that** the wood pulp derives from shoots no older than 1 year of false acacia trees, teak trees, bongassi trees or bamboo.
 3. Process in accordance with Claim 1, **characterised in that** the lignin content of the less-than-one-year-old shoots used does not exceed 7%, preferred is no more than 5%, and particularly favourable is no more than 2%.
 4. Process in accordance with Claim 1, **characterised in that** the alkali metal hydroxide solution used to treat the wood pulp in Step a) is a sodium hydroxide solution which contains between 150 and 350 g/l of sodium hydroxide.
 5. Process in accordance with Claim 4, **characterised in that** the sodium hydroxide solution contains approx. 300 g/l of sodium hydroxide.
 6. Process in accordance with Claim 1, **characterised in that** treatment of the wood pulp in Step a) is carried out at a temperature ranging between 15°C and 25°C.

7. Process in accordance with Claim 1, **characterised in that** the shredding process of the alkali cellulose in Step c) comprises a coarse comminution step and a fine comminution step.
8. Process in accordance with Claim 1, **characterised in that** the alkali cellulose crumbs in Step d) are ripened at a temperature ranging between 60°C and 80°C.
9. Process in accordance with Claim 8, **characterised in that** the alkali cellulose crumbs are ripened at a temperature of between 65°C and 75°C.
10. Process in accordance with Claim 9, **characterised in that** the alkali cellulose crumbs are ripened at a temperature of approx. 72°C.
11. Process in accordance with Claim 1, **characterised in that** the alkali cellulose crumbs in Step d) are ripened to a maturity of between 8° and 12° Hottenroth.
12. Process in accordance with Claim 11, **characterised in that** the alkali cellulose crumbs are ripened to a maturity of about 10° Hottenroth.
13. Process in accordance with Claim 1, **characterised in that** the wet sulphide process in Step e) is carried out in a solution containing carbon disulphide, sodium hydroxide and Berol.
14. Process in accordance with Claim 13, **characterised in that** the carbon disulphide content of the solution is between 150 and 250 g/l and the sodium hydroxide content is between 250 and 350 g/l.
15. Process in accordance with Claim 14, **characterised in that** the carbon disulphide content of the solution is between 180 and 210 g/l and the sodium hydroxide content is between 280 and 320 g/l.

16. Process in accordance with Claim 1, **characterised in that** subsequent ripening of the cellulose in Step g) is carried out to a maturity of between 8° and 12° Hottenroth.
17. Process in accordance with Claim 1, **characterised in that** the spinning solution downstream of the subsequent ripening of the cellulose and upstream of the filtration of the spinning solution is mixed with at least one other spinning solution produced using a process which comprises Steps a) to g) as described in Claim 1.
18. Process in accordance with Claim 1, **characterised in that** the temperature of the regenerating bath in Step i) is between 35°C and 45°C.
19. Process in accordance with Claim 18, **characterised in that** the temperature of the regenerating bath is approximately 40°C.
20. Process in accordance with Claim 1, **characterised in that** the regenerating bath in Step i) contains between 70 and 160 g/l, preferably between 90 and 140 g/l, and ideally approximately 120 g/l of sulphuric acid.
21. Process in accordance with Claim 1, **characterised in that** the regenerating bath in Step i) contains between 0.3 and 4 g/l, preferably between 0.5 and 2 g/l, and ideally approximately 1 g/l of zinc sulphate.
22. Process in accordance with Claim 1, **characterised in that** the regenerating bath in Step i) contains between 0.05 and 1 g/l, preferably between 0.1 and 0.7 g/l, and ideally approximately 0.4 g/l of Berol.
23. Process in accordance with Claim 1, **characterised in that** the spinnerets in Step i) are heated to keep them at a temperature of between 55°C and 75°C.

24. Process in accordance with Claim 23, **characterised in that** the spinnerets are kept at a temperature of between 65°C and 70°C, and preferably at approx. 67°C.
25. Process in accordance with Claim 1, **characterised in that** the spinnerets in Step i) are oval to long-slit-shaped.
26. Process in accordance with Claim 1, **characterised in that** dehydrating of the fibres in Step k) is carried out with a sulphuric acid solution which contains up to 15 g/l of sulphuric acid.
27. Process in accordance with Claim 26, **characterised in that** the sulphuric acid solution used to dehydrate the fibres contains up to 10 g/l of sulphuric acid.
28. Process in accordance with Claim 1, **characterised in that** desulphurisation of the fibres in Step l) is carried out with a sodium sulphate solution which contains between 2 and 5 g/l of sodium sulphate.
29. Process in accordance with Claim 28, **characterised in that** the sodium sulphate solution used to desulphurise the fibres contains approximately 3 g/l of sodium sulphate.
30. Process in accordance with Claim 1, **characterised in that** the twisted fibres are treated with titanium dioxide after being washed with water and before being dehydrated.
31. Process in accordance with Claim 1, **characterised in that** the predehydrating of the fibres in Step n) is carried out with compressed air.
32. Process in accordance with Claim 1, **characterised in that** the drying of the fibres in Step o) is carried out under application of tunnel dryers.

33. Cellulose fibre, obtainable by a process in accordance with one of the Claims 1 to 32.
34. Cellulose fibre in accordance with Claim 33, **characterised by** a microstructure which displays fibre-parallel lamellae.
35. Cellulose fibre in accordance with Claim 34, **characterised in that** the spacing between the fibre-parallel lamellae ranges between 1 nm and 5 μm .
36. Cellulose fibre in accordance with Claim 35, **characterised in that** the spacing between the fibre-parallel lamellae ranges between 200 nm and 1 μm .
37. Fabric comprising a backing fabric and a pile woven into the backing fabric which contains the fibres in accordance with one of the Claims 33 to 36.
38. Fabric in accordance with Claim 37, **characterised in that** the backing fabric has a lattice-like structure.
39. Fabric in accordance with Claim 37, **characterised in that** the pile forms a fibre bed of approx. 0.5 cm in height above the backing fabric.
40. Fabric in accordance with Claim 37, **characterised in that** the backing fabric contains viscose staple fibres.
41. Fabric in accordance with Claim 40, **characterised in that** the backing fabric consists exclusively of viscose staple fibres.
42. Fabric in accordance with Claim 37, **characterised in that** the pile contains oval and tape fibres.
43. Fabric in accordance with Claim 42, **characterised in that** the pile consists of 50% oval fibres and 50% tape fibres.

44. Fabric in accordance with Claim 43, **characterised in that** the pile consists of 50% of oval fibres with a count of 330 dtex F60 and 50% of tape fibres with a count of 330 dtex F80.
45. Use of the fabric in accordance with one of the Claims 37 to 44 for cleaning and decontamination applications.
46. Use of the fabric in accordance with one of the Claims 37 to 44 to reduce the surface tension of water.
47. Use of the fabric in accordance with one of the Claims 37 to 44 to make textiles.
48. Use of the fabric in accordance with one of the Claims 37 to 44 to make clothing textiles.
49. Use of the fabric in accordance with one of the Claims 37 to 44 to make personal hygiene articles.
50. Use of the fabric in accordance with one of the Claims 37 to 44 as a particle filter.
51. Use of the fabric in accordance with one of the Claims 37 to 44 as a condensation catalyst.
52. Use of the fabric in accordance with one of the Claims 37 to 44 as a floor covering.
53. Use of the fabric in accordance with one of the Claims 37 to 44 as a covering material.

002150.2228460

Abstract

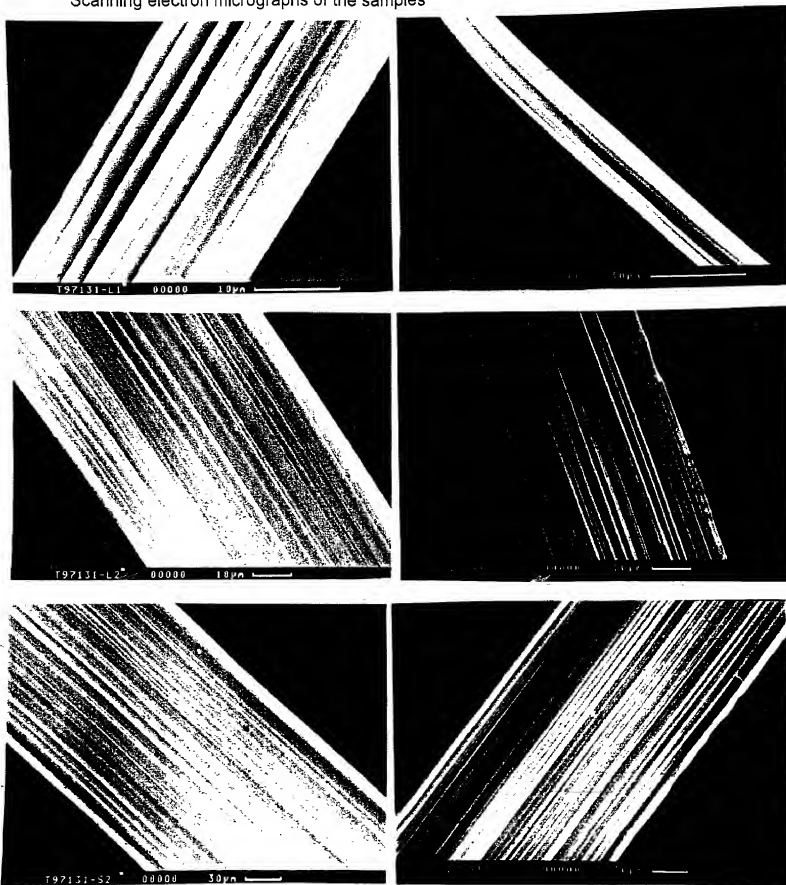
The invention described here concerns a process to permit the manufacture of a cellulose fibre from hydrate cellulose with an extremely large surface area which may be used for the preparation of a fabric characterized by a high absorptive power, good liquid-retention properties, high grease-solvent properties as well as particle-absorbing properties, which is suitable for making products that are themselves easy to clean, which can be used for cleaning and decontamination as well as to reduce the surface tension of water and which can be disposed of without damage to the environment.

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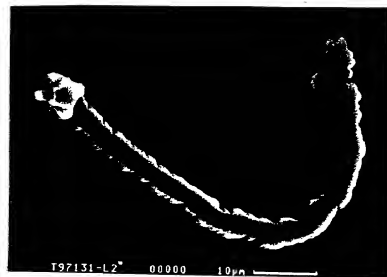
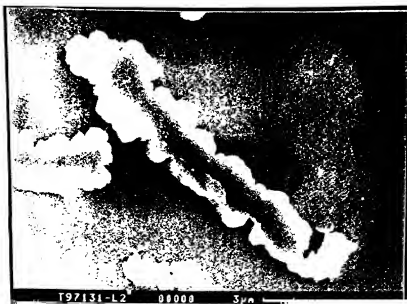
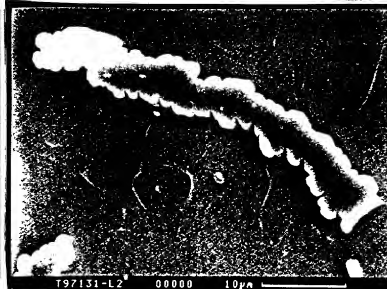
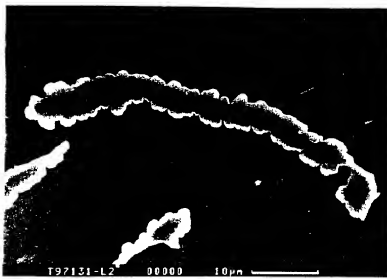
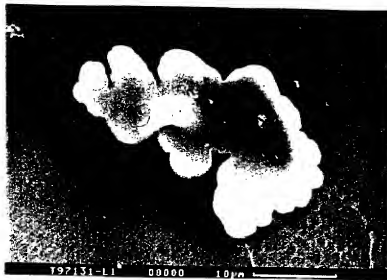
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Figures 1 to 6

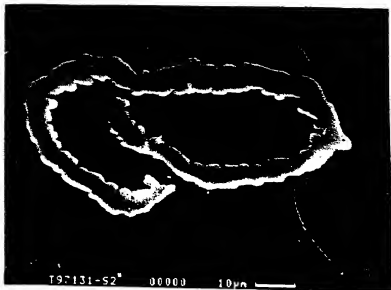
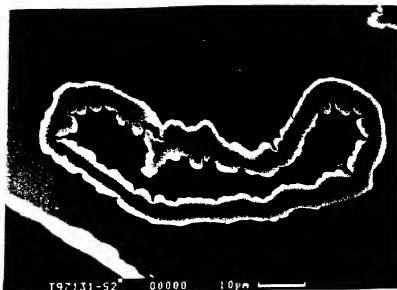
Scanning electron micrographs of the samples



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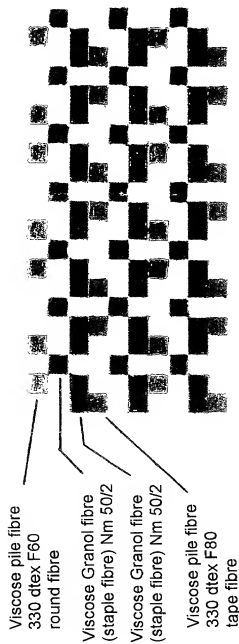
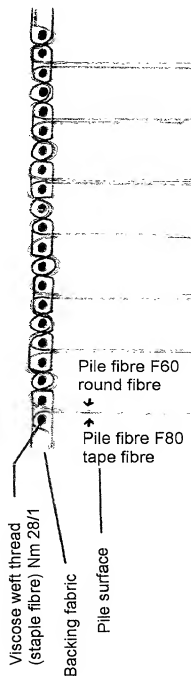
Figures 7 to 12Scanning electron micrographs of microtome sections (approx. 3 μ m thick)

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Figures 13 to 15

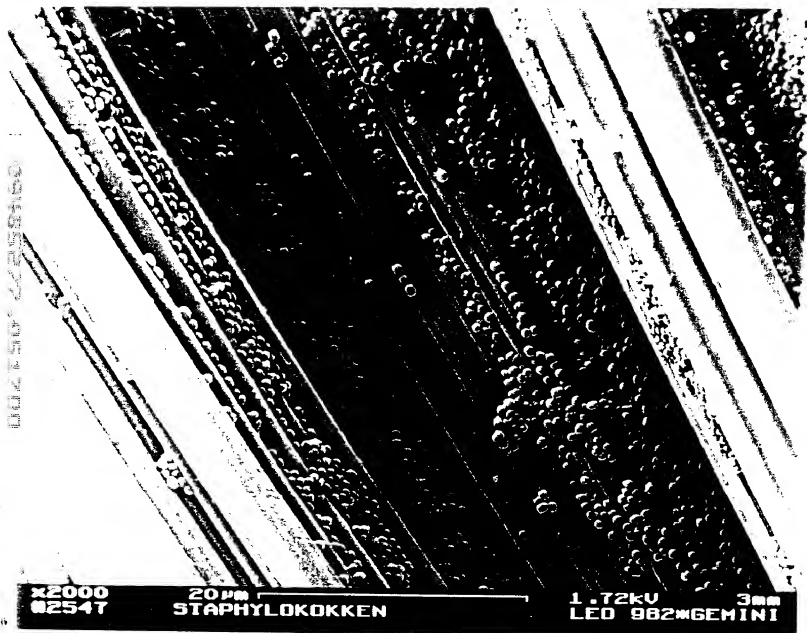
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Figure 16

Pile fabric (M-2/250)Fabric structure
(viewed from above)Fabric structure
(Section)

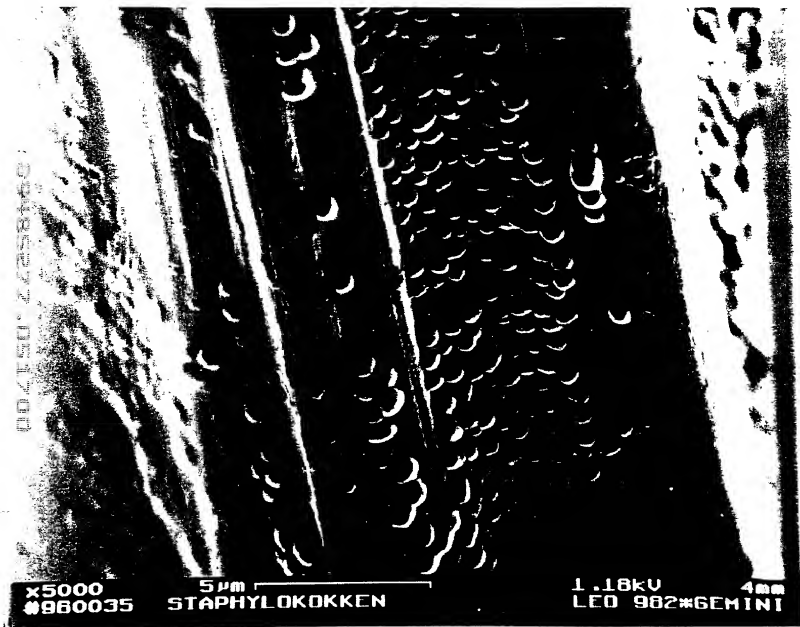
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Figure 17

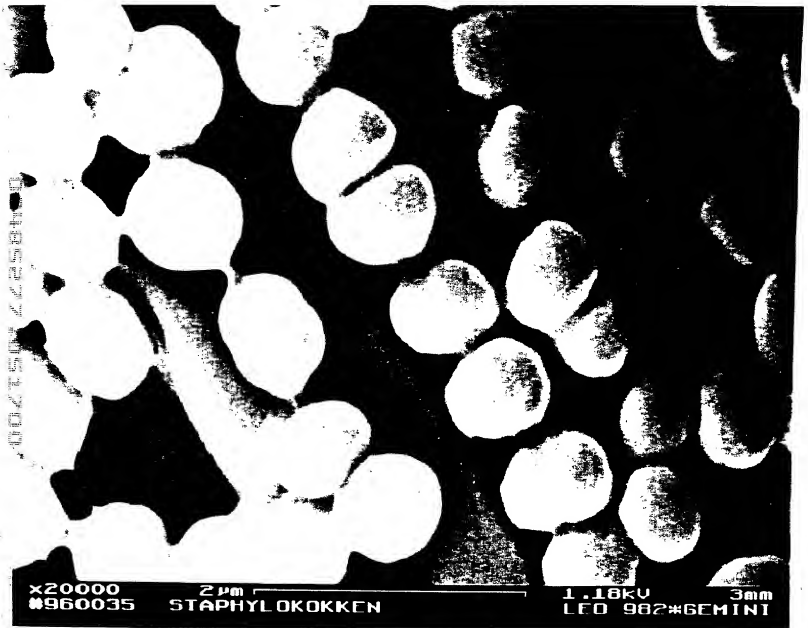


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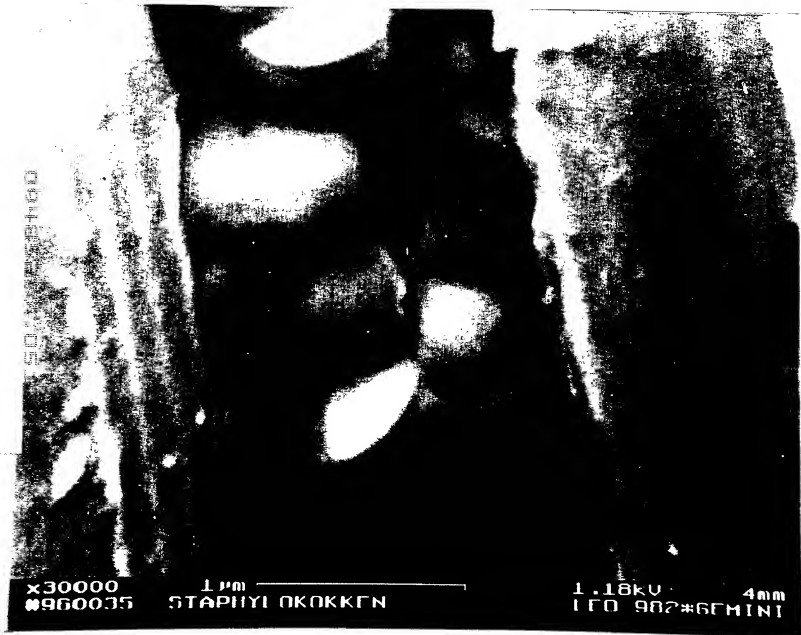
Figure 18



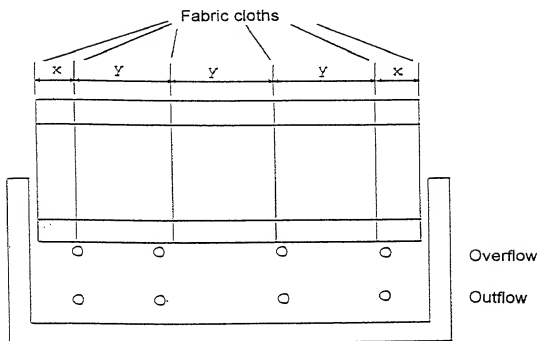
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Figure 19

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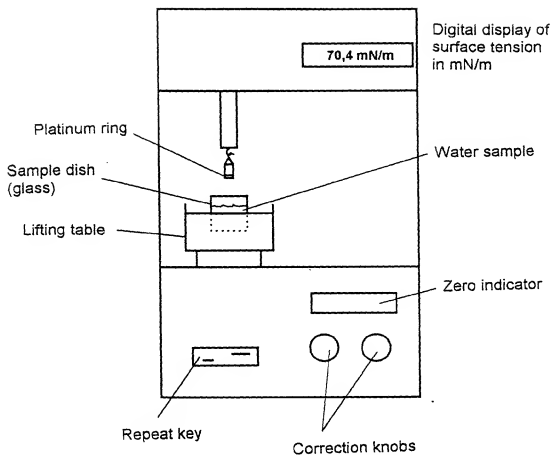
Figure 20

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Figure 21

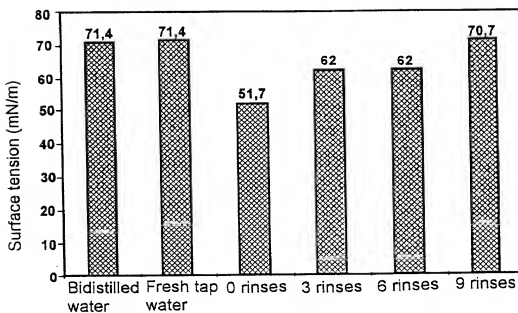
x, y: spacing between fabric cloths

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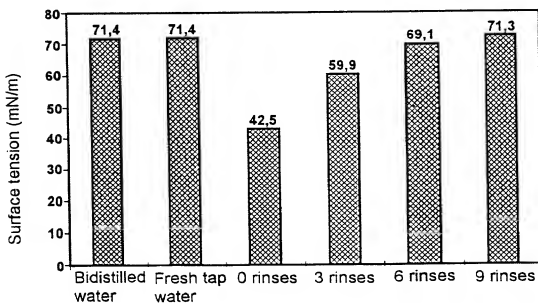
Figure 22

Tensiometer to measure the surface tension

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Figures 23 and 24

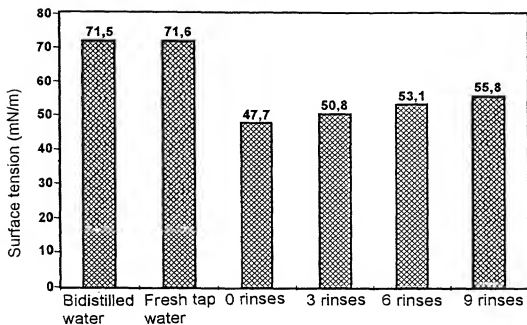
Surface tension-reducing effect of the fabric L01 as a function of rinsing the fabric



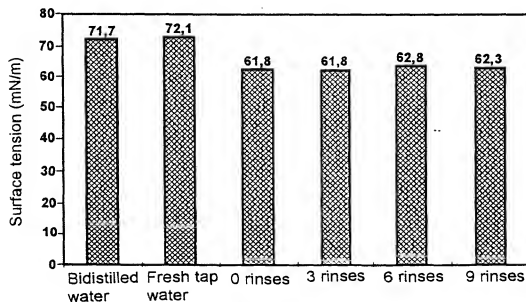
Surface tension-reducing effect of the fabric L02 as a function of rinsing the fabric

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Figures 25 and 26



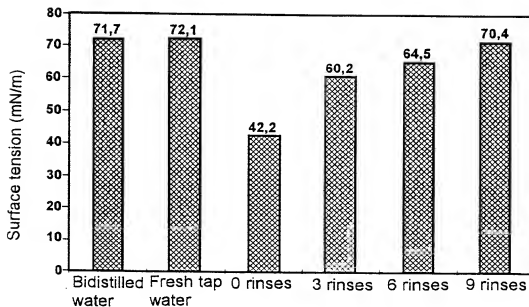
Surface tension-reducing effect of the fabric S10 as a function of rinsing the fabric



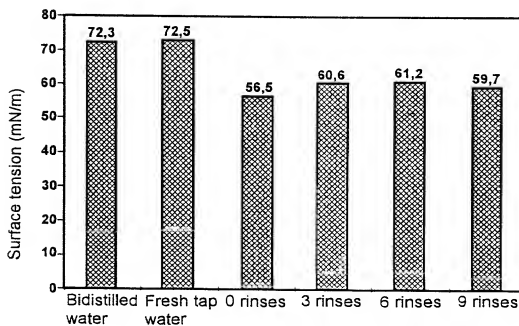
Surface tension-reducing effect of the double-sided fabric L01

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Figures 27 and 28



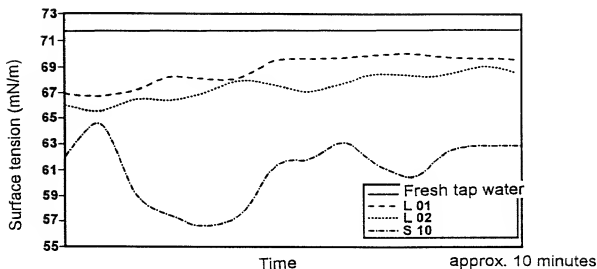
Surface tension-reducing effect of the double-sided fabric L02



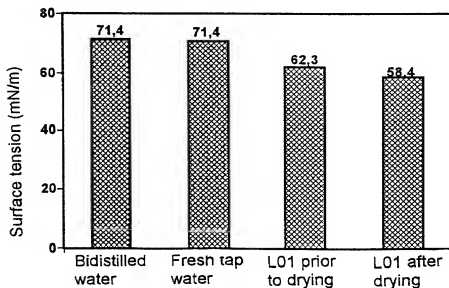
Surface tension-reducing effect of the double-sided fabric S10

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Figures 29 and 30

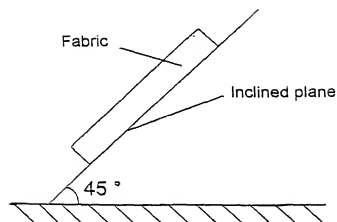


Surface tension with fabrics which remain in the water

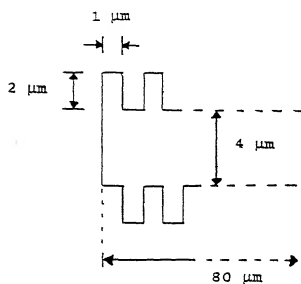


Surface tension-reducing effect of the fabric L01 before and after the drying phase (mean values)

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Figures 31 and 32

Test set-up to determine the water-absorbing capacity



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DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION (37 CFR 1.63)		Attorney Docket Number	1328/3
		First Named Inventor	Neumayr, Achim
		COMPLETE IF KNOWN	
		Application Number	09/485,277
		Filing Date	February 3, 2000
<input type="checkbox"/> Declaration Submitted with Initial Filing		Group Art Unit	
<input checked="" type="checkbox"/> Declaration Submitted after Initial Filing (surcharge (37 CFR 1.16 (e)) required)		Examiner Name	

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

PROCESS TO MANUFACTURE A CELLULOSE FIBRE FROM HYDRATE CELLULOSE

☐ the specification of which is attached hereto

OR
☒ was filed on (MM/DD/YYYY) **February 3, 2000** as United States Application Number or PCT International

Application Number: **09/485,277** and was amended on (MM/DD/YYYY) (if applicable)

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR 1.56.

I hereby claim foreign priority benefits under 35 U.S.C. 119(a)-(d) or 356(b) of any foreign application(s) for patent or inventor's certificate, or 356(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application Number(s)	Country	Foreign Filing Date (MM/DD/YYYY)	Priority Not Claimed	Certified Copy Attached?
197 34 239.6	Germany	08/07/1997	<input type="checkbox"/>	YES NO
198 09 765.4	Germany	03/06/1998	<input type="checkbox"/>	<input checked="" type="checkbox"/> <input checked="" type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>

☐ Additional foreign application numbers are listed on a supplemental priority data sheet PTO/SB/02B attached hereto:

I hereby claim the benefit under 35 U.S.C. 119(e) of any United States provisional application(s) listed below

Application Number(s)	Filing Date (MM/DD/YYYY)
	<input type="checkbox"/> Additional provisional application numbers are listed on a supplemental priority data sheet PTO/SB/02B attached hereto.

[Page 1 of 2]

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I hereby claim the benefit under 35 U.S.C. 120 of any United States application(s), or 365(c) of any PCT international application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT international application in the manner provided by the first paragraph of 35 U.S.C. 112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR 1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application.

U.S. Parent Application or PCT Parent Number	Parent Filing Date (MM/DD/YYYY)	Parent Patent Number (if applicable)
PCT/EP98/05030	August 7, 1998	

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As a named inventor, I hereby appoint the following registered practitioner(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

☐ Customer Number OR ☒ Registered practitioner(s) name/registration number listed below

Place Customer Number Bar Code Label here

Name	Registration Number	Name	Registration Number
Richard E. Jenkins	28,428	Arles A. Taylor, Jr.	39,395
Jeffrey L. Wilson	36,058	Gregory A. Hunt	41,085
Jennifer L. Skord	30,687	David P. Gloekler	41,037

☐ Additional registered practitioner(s) named on supplemental Registered Practitioner Information sheet PTO/SB/02C attached hereto.

Direct all correspondence to: ☐ Customer Number or Bar Code Label OR ☒ Correspondence address below

Name	Richard E. Jenkins, JENKINS & WILSON, P.A.				
Address	Suite 1400 University Tower				
Address	3100 Tower Boulevard				
City	Durham	State	NC	ZIP	27707
Country	USA	Telephone	001-919-493-8000	Fax	001-919-419-0383

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Name of Sole or First Inventor:		<input type="checkbox"/> A petition has been filed for this unsigned inventor			
Given Name (first and middle (if any))		Family Name or Surname			
Achim		Neumayr			
Inventor's Signature	<i>Achim Neumayr</i>		25.04.00	Date	
Residence: City	Villenbach	State		Country	Germany
				Citizenship	German
Post Office Address	Demhartstrasse 8				
Post Office Address					
City	Villenbach	State		ZIP	D-89444
				Country	Germany

☒ Additional inventors are being named on the 1 supplemental Additional Inventor(s) sheet(s) PTO/SB/02A attached hereto

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DECLARATION

ADDITIONAL INVENTOR(S) Supplemental Sheet

Page 1 of 1

Name of Additional Joint Inventor, if any:				<input type="checkbox"/> A petition has been filed for this unsigned inventor			
Given Name (first and middle (if any))				Family Name or Surname			
Herbert				Hael			
Inventor's Signature	<i>J. Van</i>			25.04.00	Date		
Residence: City	Oberammergau	State		Country	Germany	Citizenship	German
Post Office Address	Puerschlingweg 4 <i>DGL</i>						
Post Office Address							
City	Oberammergau	State		ZIP	D-82487	Country	Germany
Name of Additional Joint Inventor, if any:				<input type="checkbox"/> A petition has been filed for this unsigned inventor			
Given Name (first and middle (if any))				Family Name or Surname			
Inventor's Signature				Date			
Residence: City		State		Country		Citizenship	
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Post Office Address							
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